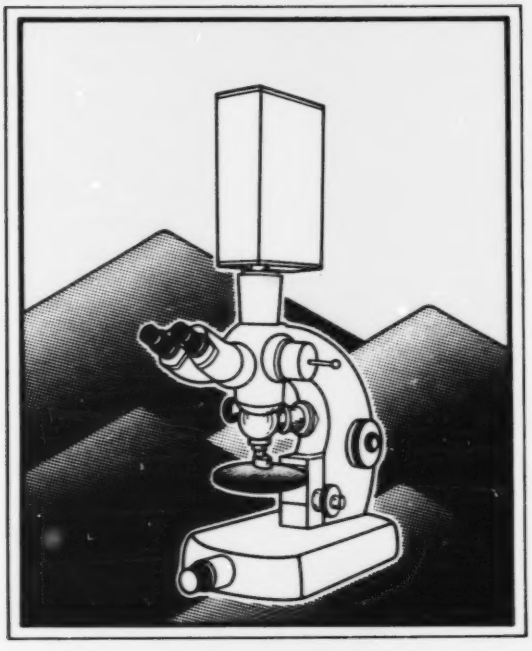
Information Series 102

Application of coal petrography in the liquefaction of subbituminous coals and lignites



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1983

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Table of Contents

Summary 1
Introduction 2
Part I
Fundamentals of coal petrology 2
  Description of macerals 3
Sample preparation 4
Reflectance measurement 5
Procedure of maceral analysis 6
Petrography of coal liquefaction residues 6
  Classification of residue components 6
Part II
Petrographic composition 8
  Petrographic analysis of subbituminous coals and lignites 8
  Reflectance of the vitrinite (huminite) component 11
  Reflectance versus chemical composition 12
  Photomicrographs 16
Petrographic composition of density fractions 16
Liquefaction behavior and maceral composition 20
Petrographic analysis of liquefied coal residues 22
References 24

Tables

Table 1. Strippable coal reserves in the plains region of Alberta 2
Table 2. Classification of macerals 3
Table 3. Relationship between coal rank and approximate reflectance limits 5
Table 4. Chemical composition of coals 9
Table 5. Petrographic analyses of Alberta Plains coals 9
Table 6. Chemical composition of lignites 10
Table 7. Petrographic analysis of lignites 10
Table 8. Comparision of petrographic analyses of a subbituminous coal 10
Table 9. Random oil reflectance of subbituminous coals and lignites 12
Table 10. Chemical analysis of density fractions of U.S. lignites 17
Table 11. Chemical analysis of density fractions of subbituminous coals 17
Table 12. Maceral analysis of density fractions of North Dakota lignite 18
Table 13. Maceral analysis of density fractions of Texas lignite 18
Table 14. Maceral analysis of density fractions of Highvale coal (subbituminous B) .. 19
Table 15. Maceral analysis of density fractions of Vesta mine coal (subbituminous C) 19
Table 16. Liquefaction behavior dependence on coal characteristics 20
Table 17. Petrographic analysis of liquefied coal residues 22

Figures

Figure 1. Coalification tracks of different macerals based on H/C:O/C atomic ratios . . 3
Figure 2. The reflectance of three macerals at different levels of rank 5
Figure 3. Possible alterations of vitrinite maceral in liquefaction 7
Figure 4. Alberta coal deposits: location of coals analyzed 8
Figure 5. Relationship between vitrinite-liptinite macerals and H/C atomic ratio 10
Figure 6. Relationship between vitrinite-liptinite macerals and O/C atomic ratio 11
Figure 7. Relationship betwen fusinite-semifusinite macerals and H/C atomic ratio . . 11
Figure 8. Relationship between fusinite-semifusinite macerals and O/C atomic ratio . 11
Figure 9. Reflectance histogram of Smoky Tower coal 13
Figure 10. Reflectance histogram of Highvale coal 13
Figure 11. Reflectance histogram of Vesta mine coal 13
Figure 12. Reflectance histogram of North Dakota lignite 13
Figure 13. Reflectance histogram of Texas lignite 13
Figure 14. Reflectance percent distribution curve of Smoky Tower coal 14
Figure 15. Reflectance percent distribution curve of Highvale coal 14
Figure 16. Reflectance percent distribution curve of Vesta mine coal 14
Figure 17. Reflectance percent distribution curve of North Dakota lignite 14
Figure 18. Reflectance percent distribution curve of Texas lignite 14
Figure 19. Reflectance frequency distribution diagram for subbituminous coals 15
Figure 20. Reflectance percent distribution curve for subbituminous coals 15
Figure 21. Reflectance frequency distribution diagram for U.S. lignites 15
Figure 22. Reflectance percent distribution curve for U.S. lignites 15
Figure 23. Relationship between mean reflectance of vitrinite and carbon content 16
Figure 24. Schematic procedure for separation of density fraction of lignite 16
Figure 25. Solubilities of North Dakota lignite 20
Figure 26. Solubilities of Texas lignite 20
Figure 27. Relationship between huminite contents and lignite solubility 21
Figure 28. Behavior of huminite and vitrinite macerals and submacerals upon hydrogenation 21

Plates 26

Summary

This work systematically scans Alberta Plains coals from the major coal regions for their petrographic composition and develops methods for petrographic analysis of solid residues from liquefaction. The objectives of these studies are to gain a better understanding of how coal structure changes under different liquefaction process conditions, and ultimately, to develop a means of predicting product yields starting from the rank and maceral composition of the raw coal.

Part I of this report gives a general introduction to coal petrology and its application in coal liquefaction. Part II describes the petrographic composition of some Alberta subbituminous coals and their residues from liquefaction.

Ten samples of coal from different regions are analyzed for their petrographic composition. In addition, for comparison, two U.S. lignites (Texas and North Dakota) and two Saskatchewan lignites are included in these investigations. In maceral analysis, fluorescent-light microscopy was used to identify auto-luminescent liptinitic macerals. The data make a point that vitrinitic and liptinitic macerals, which account for more than 75 percent of the total organic macerals, could be good diagnostic indicators for the chemical parameters (C, H and O) of subbituminous coals and lignites. Alberta subbituminous coals have low concentrations of the macerals of the liptinitic group and are almost free from pyrite.

Based on reflectance of vitrinite, the data match ASTM rank classification for subbituminous coals. Among the lignites, the degree of coalification based on reflectance of huminite (vitrinite) follows the order: Saskatchewan lignites—Texas lignite—North Dakota lignite

Texas lignite is richer in liptinite but poorer in semifusinite than North Dakota lignite. Both subbituminous coals and lignites show a good relation of reflectance of vitrinite (huminite) to carbon content.

Paucity of the liquefaction data, in terms of number of subbituminous coal samples at this stage, does not permit development of mathematical correlation between liquefaction conversion yields and petrographic composition. On plotting total liquefaction conversion of lignites and their density fractions against their huminite (vitrinite) contents, however, a rectilinear correlation becomes apparent. It is premature at this stage to advocate any conversion factor for any maceral group to predict the liquefaction behavior of a subbituminous coal or lignite.

Petrographic analysis of residues produced during coal liquefaction appears to be quite useful in providing new insights into the process of liquefaction. Optical examination allows identification of four main categories of residue components:

(i) relatively unaltered components;
(ii) thermally altered components (vitroplast and cenospheres);
(iii) granular residue; and
(iv) inorganic components.

Vitrinite (huminite) and liptinite group macerals are the principal reactive constituents of coals. Vitrinite gives rise to several products in the solid residues of liquefaction. Semifusinite solubilizes partially and the unreacted portion appears in the residue. It undergoes mechanical grinding and as a result distinction between semifusinite and fusinite cannot be obtained. Further contributing to the difficulty in distinguishing semifusinite and fusinite is enhanced reflectance of semifusinite during the liquefaction process. Fusinite merges with granular residue after simple mechanical fining and remains undistinguished. Semicoke originates from liquefied vitrinite under severe reaction conditions of liquefaction. It would be premature to evaluate with confidence the liquefaction behavior of a particular subbituminous coal from the comparative study of maceral composition with those of the liquefaction residues.

Introduction

Alberta's deposits of subbituminous coal represent a large potential source of liquid fuels. Surface mineable coal in the Plains Region of Alberta is the most attractive feedstock for future liquefaction plants. The Energy Resources Conservation Board (1981) estimates 6.7 gigatonnes of strippable coal reserves (table 1). If oil prices continue to increase and an efficient conversion technology is developed, coal could compete economically with oil sands as a source of synthetic liquids.

The chemical composition of Alberta Plains coals is well known; the petrographic characteristics are not. Coal is an extremely heterogeneous mixture of various mineral phases and macerals. The application of coal petrography to technical processes such as coal liquefaction, carbonization and combustion could be aimed at developing, improving or controlling such processes. It is not known what characteristics (petrographic or chemical) are important in determining coal conversion reactivity.

This work systematically scans Alberta Plains coals from the major coal regions for their petrographic composition and develops methods for petrographic analysis of solid residues from liquefaction. The ultimate aim of these studies is to predict the yield of liquid and gaseous products, starting from the given rank of the coal and a given maceral composition.

Until recently, no systematic research was undertaken to study the morphology and the nature of liquefied coal solid residues or to make comparisons with the petrography of the feed coal. Petrographers from different countries have just started an intensive research program, observing changes in the nature and composition of residues produced during small-scale hydrogenation at progressively increased temperatures, pressures or residence time in the reaction (Mitchell *et al.*, 1977; Shibaoka, 1978; Shibaoka and Ueda, 1978; Wakeley, *et al.*, 1979; Diessel, 1979; Barras *et al.*, 1979). Preliminary investigations indicate that in high volatile coals used for liquefaction, macerals of vitrinite and liptinite groups are regarded as more or less reactive, whereas inertinite macerals are to be subdivided into low reflecting types (R = < 1.5 percent) — reactive to a certain extent, and high reflecting types (R = > 1.5 percent), which are always inert.

Table 1.
Strippable coal reserves in the plains region of Alberta

Strip ratio cu m/tonne	Reserves gigatonnes	Percentage of total
0 to 5	1.5	22.4
5 to 10	3.3	49.2
10 to 15	1.7	25.4
>15	0.2	3.0
Total reserves	6.7	100.0

Part I
Fundamentals of coal petrology

Optically homogeneous organic materials in coal are called macerals. They are identified and classified on the basis of their morphology, source material, color or level of reflectivity, and nature of formation. There are three major groups of macerals:

a. Vitrinite ('huminite' is restricted to low rank coals —lignites and subbituminous);
b. Liptinite (exinite);
c. Inertinite.

Table 2 gives the classification of macerals. Each group includes a series of macerals that have either a similar origin or the same mode of conversion from original plant material to coal. The three maceral groups are, to some degree, characterized by their chemical composition. Comparing groups of the same rank shows that the vitrinite contains relatively more oxygen, the liptinite more hydrogen and the inertinite more carbon. Figure 1 shows the composition of different macerals based on H/C and O/C atomic ratios (van Krevelen, 1962).

Lignites have a much greater degree of petrographic heterogeneity than coals. This is to be expected since

Table 2. Classification of macerals

Group	Maceral
Vitrinite	Telinite Collinite "Pseudovitrinite" Vitrodetrinite
Liptinite (exinite)	Sporinite Cutinite Resinite Alginite Suberinite Liptodetrinite
Inertinite	Fusinite Semifusinite Inertodetrinite Macrinite Micrinite Sclerotinite

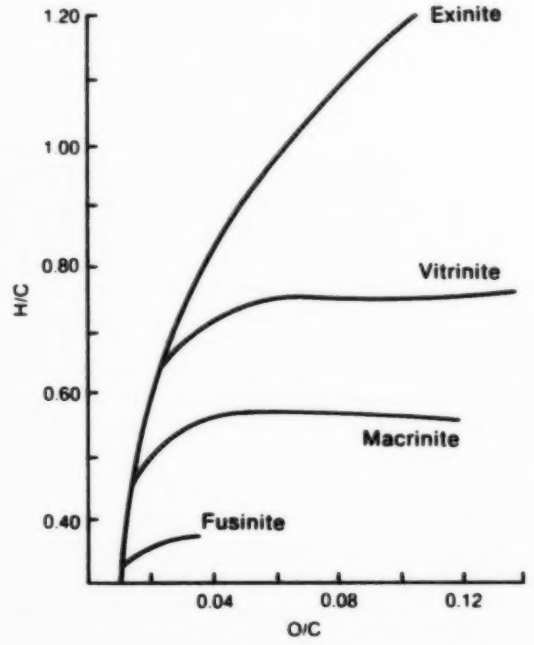


Figure 1. Coalification tracks of different macerals based on H/C:O/C atomic ratios

the vegetal materials in lignites have been altered to a far lesser degree than in higher rank coals. The nature and properties of some petrographic entities in lignites are different from those of the macerals of hard coals. Basically, the huminite group in lignites is equivalent to, and the precursor of, the vitrinite group of hard coals.

The reflected light method is the most widely used petrographic analysis technique because of the relative ease in sample preparation. The technique requires that the coal sample be optically polished and free of scratches. The samples are studied in an oil immersion with the aid of a reflected light microscope. The oil immersion technique allows a better resolution of polished surface, a more accurate quantitative count of macerals and better reflectance measurement of the macerals. A microscope equipped with a vertical illuminator and oil immersion objectives in the 40 to 60 magnification range is adequate for maceral analysis.

Description of macerals

A brief description of the macerals and maceral groups identified (see plates 1 to 5) in this study follows:

Vitrinite group

Vitrinite is the most abundant and most important maceral in coals, accounting for, in most instances more than 70 to 80 percent of any given coal bed. In reflected light, vitrinite appears to be dark gray, light gray, or white, depending on the rank. The density varies between 1.3 to 1.8 gcm^{-3}, and the pore volume is in the range 0.03 to 0.05 $cm^3\ g^{-1}$.

Liptinite (exinite) group

The macerals of the liptinite group are derived from the resinous and waxy material of plants, and have the highest hydrogen content among all macerals (Ting, 1972). Liptinites are dark gray to gray in reflected light. Individual macerals of the liptinite group are classified primarily on the basis of their morphology (table 2). To identify the macerals of liptinite, fluorescent light is usually recommended. Liptinite is the lightest component of the coal and the density ranges between 1.18 to 1.28 gcm^{-3} for bituminous coals. Its toughness contrasts with the brittleness of vitrinite and in low rank coals the index of reflectivity is very low compared with those of other macerals.

Inertinite group

Inertinites are the group of macerals that are bright in reflected light. Inertinite derives its name from the fact that it is inert or semi-inert during the normal carbonization process. Depending on their shape, size, degree of preservation of cellular structure, and reflectance, inertinites are subdivided into fusinite, semifusinite, inertodetrinite, macrinite, micrinite and sclerotinite.

Fusinite group

Fusinite maceral is the richest in carbon of all the constituents of coal. In general, its proportion is low and does not exceed a few percent. In reflected light its color is yellowish-white to white and the reflectance is high. It is not true that the reflectance of fusinite is always the highest of all macerals (Alpern and De Sousa, 1970). The density of fusinite, about 1.5 gcm $^{-3}$, exceeds that of vitrinite and liptinite. During carbonization, the fusinite does not fuse and physico-chemical properites in general do not vary much with increasing rank.

Semifusinite group

This transitional stage between fusinite and telinite (vitrinite group maceral) always shows reflectance intermediate between vitrinite and fusinite and is of white or light gray color. The physical and chemical properties of semifusinite vary widely, ranging from those of vitrinite to those of fusinite, according to its degree of coalification. Semifusinite is richer in carbon and poorer in hydrogen than vitrinite, but poorer in carbon and richer in hydrogen than fusinite. The density varies from about 1.35 to 1.45 gcm $^{-3}$.

Inertodetrinite is made up of strongly reflecting fine particles (normally less than 30 μm) and is believed to derive from fragmented fusinite and semifusinite.

Macrinite is a more or less amorphous, non-granular groundmass of high reflectance and is a relatively rare maceral. Micrinite is characterized by the rounded shape, the very small size (one μm) of its grains, and higher reflectance than the associated vitrinite.

Sclerotinite is recognized as fungal remains. The reflectance is commonly high and it is assumed that it contains much carbon and little hydrogen. The density of sclerotinite from soft brown coals (equivalent to subbituminous coals) varies between 1.6 to 2.0 gcm $^{-3}$.

Sample preparation

The principal objective of the sample preparation procedure is to prepare, for microscopic examination, a suitable polished surface which is representative of the coal under consideration. This usually invoives crushing the coal, embedding it in a mounting medium, and grinding and polishing a surface, which should meet the following requirements:

a. The entire surface should be flat, and with little or no relief between the various coal components, or at the edges of coal particles.
b. It should be free of pits caused by loss of coal grains.
c. It should appear substantially free of scratches under the microscope.
d. It should be clean and free of grinding and polishing compounds.

For reflected light examination, coal samples are usually prepared as briquets of crushed particles using epoxy resin as binding medium. The crushed coal (less than 0.850 mm size) is mixed thoroughly with an epoxy resin mixture in the ratio of about four parts of coal to one part epoxy by volume in a cylindrical plastic mould, 2.54 cm in diameter, which is subsequently filled to the top with an epoxy resin mixture. The mixture is then allowed to cure in the mould overnight. After removal from the mould, the briquets are placed in a holder and are ready for grinding and polishing with an automated grinder/polisher. For detailed procedures, reference should be made to ASTM Standard D-2797 (1975).

The moisture content can significantly affect the reflectance values of some coals (Harrison, 1965, 1966). Consequently, pellets should be kept in a desiccator for fifteen hours prior to measurement. However, in the case of low rank coals, which tend to slack under dry conditions, careful compromise is needed for these coals (Davis, 1978). Oil reflectance limits as guides to coal ranks are given in table 3.

Reflectance measurement

Reflectance is an index of the degree of metamorphism of coal. Reflectance is defined as the proportion of normally incident light that is reflected by a plane, polished surface of the substance under consideration. It is usually expressed as a percentage, so that reflectance of 1.00 percent for vitrinite indicates that only that proportion of the light incident on the coal surface is reflected under the conditions of measurement.

For most purposes, the reflectance of the vitrinite maceral of coal is measured because:

a. Vitrinite is the most abundant maceral in most coals.
b. Vitrinite often appears homogeneous under the microscope.
c. Particles of vitrinite are usually large enough to permit measurement to be made easily.
d. The reflectance of vitrinite varies more uniformly than that of other macerals as coal rank increases.
e. In the application of petrographic techniques for the industrial uses of coal, particularly in carbonization, interest is focused on the behavior of vitrinite, which is the maceral principally responsible for the plastic and agglutinating properties of coal.

Measurement of the reflectance of the coal maceral vitrinite has become a routine procedure in characterization of coal rank. Rank determination is performed by measuring either maximum reflectance in polarized, reflected light, or random (average) reflectance, usually in non-polarized reflected light. Choice between the two techniques depends upon individuals. Measuring maximum reflectance requires that the object stage be rotated until a maximum value of reflectance is recorded.

Points favoring the use of random reflectance are the time consumed in rotation of the microscope stage is avoided and the measurement avoids error due to lack of centering and change in focus on rotation (Davis, 1978).

The reflectance of coal has proved to be a rapid and accurate index of coal rank, and is largely uninfluenced by the petrographic variation that can affect the results of many chemical tests used for the same purpose. One principal advantage is that reflectance can be made on extremely small amounts of material. It is also a non-destructive technique and measurements can be repeated on the same sample. Few data have been published on the reflectance of macerals other than vitrinite. Figure 2 shows the relative reflectance of vitrinite, exinite, and micrinite at different carbon contents of the vitrinites (Dormons *et al.*, 1957). Fusinite is generally the highest reflecting maceral.

Table 3.
Relationship between coal rank and approximate reflectance limits*

Coal rank		% Reflectance (maximum)
Lignite		< 0.27-0.38
Subbituminous	C	0.38-0.43
	B	0.43-0.48
	A	0.48-0.67
High volatile bituminous	C	0.47-0.57
	B	0.57-0.71
	A	0.71-1.10
Medium volatile bituminous		1.10-1.50
Low volatile bituminous		1.50-2.05
Semianthracite		2.05-3.00 (approximately)
Anthracite		3.00 (approximately)

*from Davis (1978) and Stach *et al.* (1982)

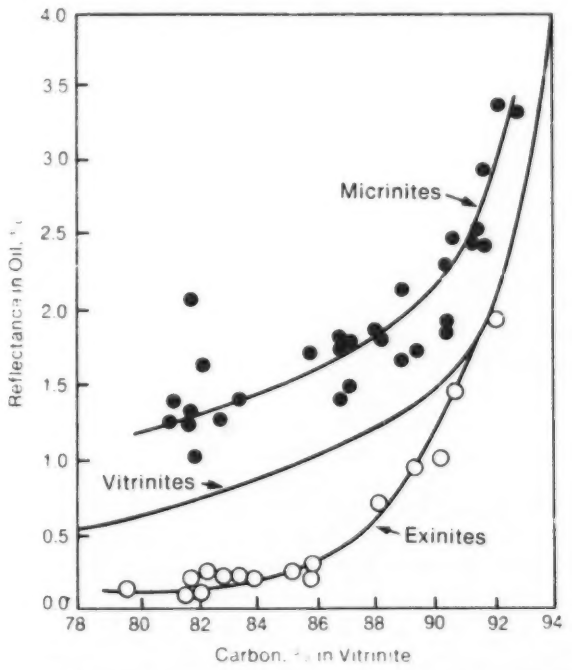


Figure 2. The reflectance of three macerals at different levels of rank

Procedure of maceral analysis

In routine petrographic analysis, relative abundances of macerals are obtained by the point count method, reported as volume percent. Within each field of view, the maceral under each cross-hair of the eye piece reticle is identified and counted, normally 5 to 10 points in each field. A total of about 500 counts are made. The following macerals are usually reported: vitrinite, liptinite (including sporonite and resinite), micrinite, macrinite, inertodetrinite, fusinite and semifusinite. Petrographic composition of coals can vary a great deal, greatly affecting the fixed carbon content or calorific value of coal (Ting, 1972; Tschamler and de Ruiter, 1966; Babu and Dutcher, 1966). High inertinite content will cause an increase in fixed carbon content of a coal, and high liptinite content will effectively increase the calorific value of a coal.

In maceral analysis, advantage can be taken of the fact that part of the macerals, in particular liptinite, are auto-luminescent when irradiated with long-wave ultraviolet or blue light. The intensity and color of the fluorescence depend on the types of liptinitic macerals, rank and the degree of biochemical degradation. In low rank coals (lignite and subbituminous coal), the fluorescence of resinite and alginite usually appears greenish, whereas sporinite and cutinite appear yellowish. As rank increases, fluorescent color changes from yellowish orange to brown.

In recent years, fluorescent-light microscopy has proved very useful for studies on coal and this method of examination holds great potential to be used for routine petrographic analyses. In peat and subbituminous coals, liptinite exhibits a fluorescence of high intensity (Jacob, 1964) which, however, decreases with increasing rank. In lignite, not only the liptinite, but also some of the macerals of the huminite group exhibit fluorescence. In contrast to these, many vitrinites of bituminous coals do not fluoresce. With the exception of some semifusinites, the macerals of the inertinite group do not fluoresce at all irrespective of rank.

Petrography of coal liquefaction residues

Coal petrographic studies have, in recent years, provided new insights into the process of liquefaction. Studies that have been particularly rewarding in following progressive degradation and reactions are those of residues obtained from autoclave batch tests performed over a range of temperatures.

The formation of coke has been shown to be influenced by the proportions and the manner of association of reactive macerals and inerts in the coal. This, together with the variable properties of semifusinite have made necessary a reassessment of the role of inertinite in coal conversion processes. Comparative study of maceral composition and rank of raw (feed) coals with those of the relevant hydrogenation residues enable conversion rates and efficiencies to be assessed for the various coals. This approach, together with a knowledge of the behavior of coal macerals in other thermal processes, is enabling the individual components of residues to be related to their precursor macerals in the feed coals (Mitchell *et al.*, 1977). With the aid of optical microscopy, as with coke making, one may soon be able to predict, with some confidence, the outcome of hydrogenating a particular coal under specific conditions.

Classification of residue components

Mitchell *et al.* (1977) proposed a classification of residue components derived from the liquefaction of coals. Four main categories of residue components are identified.

Relatively unaltered components

These are macerals that have passed through the hydrogenation process with little or no alteration. Identification is commonly easy because the features are similar to those of the original coal maceral, although reflectance is increased in many instances. Fragments of vitrinite in residues from the subbituminous coal can still display a cell structure that enables the particular maceral precursor to be identified. Also, fragments of inertinite can be identified. Some of the original particle integrity is maintained.

Thermally altered components

These residue components of organic origin have

undergone a phase change. Their optical characteristics differ from those of the coal entities from which they have been derived. Nearly all of these materials have had vitrinites as their precursor. Materials and structures that constitute the plastic residue components of coal are vitroplast and cenospheres.

Vitroplast: When vitrinite from bituminous coals is heated, it may become plastic at a temperature of about 350 °C. This component is termed vitroplast. The circumstances of vitroplast formation during the liquefaction of coals seems to be similar to the development of what has been termed metaplast during the carbonization process. Vitroplast-like materials in the residues of subbituminous coals are believed to have been produced by a hydrogenative transformation of the vitrinite macerals rather than through simple thermoplastic softening.

Cenospheres: Cenospheres are globular structures that may or may not be filled and often have a reticulated internal texture. Their development can take place as an immediate result of vitroplast formation. Cenospheres are believed to result from gas production by thermal cracking. Davis *et al.*, (1967) and Neavel (1975) reported cenosphere formation during the solvation process. According to Neavel (1975), cenospheres disintegrate during the solvation process.

'Semicoke' can form as a result of exposure to elevated temperatures of the solid products of liquefaction. In fact, semicoke may originate from semifusinite (non-plastic components), vitroplast and cenospheres.

Granular residue

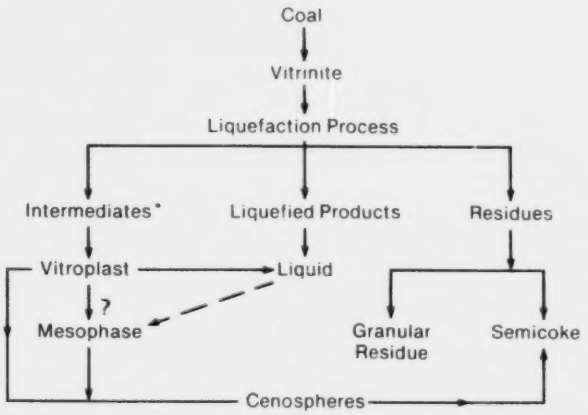
Granular residue is a submicron granular material derived from the physical dispersion of clay minerals and possibly micrinite, and the insoluble residue following the dissolution of vitrinite and liptinite. Only a small percentage of the granular residues is organic and the remainder is presumed to be clay mineral particles.

Inorganic components

Microscopically recognizable entities present in liquefaction residues are:

a. those such as quartz, that remain physically and chemically unaltered through the process;
b. those such as clay minerals, that have undergone extensive physical breakdown during the process;
c. those that have undergone major chemical and crystallographic transformations, such as conversion of pyrite to pyrrhotite;
d. those minerals, such as calcium carbonate, that form as a result of the process conditions, which crystallizes in significant amounts during solvent-refined-coal production.

Possible alterations of vitrinite macerals in liquefaction are summarized in figure 3.



*A portion of the intermediates (Vitroplast and Cenosphere) appear as residues.

Figure 3. Possible alterations of vitrinite maceral in liquefaction

The rank and petrographic composition of coals represent fundamental characteristics that determine behavior in liquefaction processes. Vitrinite and liptinite group macerals are the principal reactive constituents of coals and are, therefore, the main parent materials of liquid and gaseous products. Vitrinite gives rise to several products that are in the solid residues of liquefaction. Process conditions are important to determine which, if any, of the reactive-derived solids will be produced. Provisional results seem to indicate that:

a. relatively unaltered macerals will appear in the residue if temperatures are too low for efficient conversion;
b. highly reflecting cenospheres and semicokes can be produced if reaction conditions are too severe;
c. semicoke may form if temperatures, coal feed rate and inert content of coal are too high and there is low availability of transferable hydrogen from the solvent.

Part II
Petrographic composition

A coal research program was started in 1980 in the Alberta Research Council to evaluate Alberta subbituminous coals for liquefaction, pyrolysis and combustion behavior. In this program, ten samples of coal from different regions (figure 4) were analyzed systematically for their petrographic composition. These include three reference coals selected for detailed liquefaction studies on the basis of accessibility, and represent the complete range of geographic, rank and geological variation present in the Plains Coal Region. These deposits represent the three ASTM subbituminous ranks: subbituminous A (Smoky Tower), subbituminous B (Highvale-Wabamun) and subbituminous C (Vesta-Central Battle River). Homogenized bulk samples representative of the recoverable coal deposits from the respective regions were obtained by a bucket auger drill rig. Samples are stored in sealed barrels at low temperatures to minimize long-term deterioration.

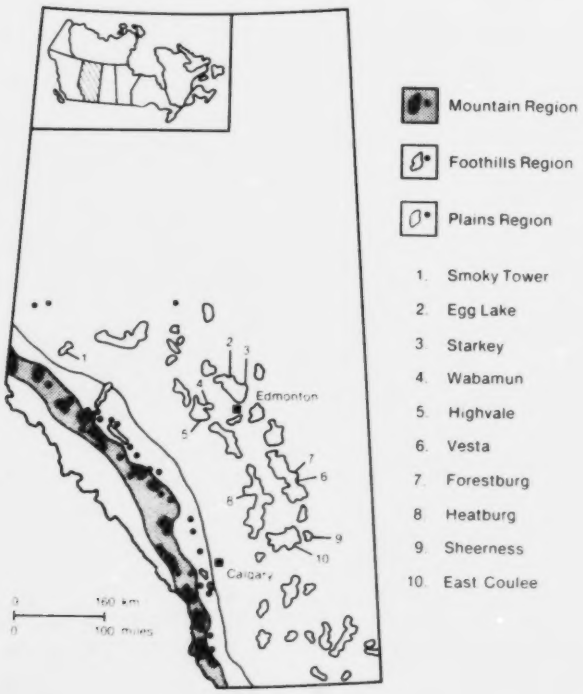


Figure 4. Alberta coal deposits: location of coals analyzed

For comparison, two U.S. lignites (Texas and North Dakota) and two Saskatchewan lignites are included in these studies.

Petrographic analysis of subbituminous coals and lignites

Chemical composition and petrographic composition of ten subbituminous coals and four lignites are given in tables 4 through 7. Data reported for petrographic composition (tables 5 and 7) are based on 900 points and are the average of three analyses by three operators on the same polished surface. To show how the analyses made by different operators vary, maceral composition for Smoky Tower (subbituminous A) coal repeated by five operators are given in table 8. For all the subbituminous coals and lignites, results seldom differed by more than five percent. The identification of macerals in lignites and subbituminous coals is far more complicated than in bituminous coals. Until fairly recently, subbituminous coals and lignites have received less attention from petrographers than have higher rank coals. There are unavoidable errors in identification of macerals of low rank coals, and there is a lot of scope for improvement in the techniques applied presently.

Each maceral of coal varies widely in its chemical composition and physical properties (Zweitering and van Krevelen, 1954). With increasing rank, the macerals vary as their chemical, physical and technological properties alter. To establish the existence of a distinct relation, if any, between maceral composition and chemical composition in the subbituminous coals and lignites, volume percent of vitrinite plus liptinite and fusinite plus semifusinite versus H/C and O/C atomic ratios were plotted (figures 5 through 8). The data indicate only a loose correlation between vitrinite plus liptinite contents and H/C atomic ratios, the concentration of fusinite plus semifusinite does not correlate in any manner to the chemical composition of subbituminous coals and lignites under investigation. These observations make a point that vitrinitic and liptinitic macerals, which account for more than 75 percent of the total organic macerals, could be relatively good diagnostic indicators for the chemical parameters (C, H, and O) of subbituminous coals and lignites.

Table 4. Chemical composition of coals

Sample no.	Location	ASTM rank	Proximate analysis (%)				Ultimate analysis (%) (moisture & ash-free)				
			Moisture	Ash (dry)	Volatile matter (d.a.f.)	Fixed Carbon (d.a.f.)	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen
1	Smoky Tower	Sb A	8.9	20.0	43.4	56.6	75.8	5.1	1.7	0.5	16.9
2	Egg Lake	Sb C	16.4	14.6	43.2	56.8	74.2	4.5	1.6	0.4	19.3
3	Starkey Mines	Sb C	25.3	9.5	42.3	57.7	74.3	4.9	1.5	0.4	18.9
4	Wabamun	Sb B	7.3	12.5	39.6	60.4	73.3	4.3	1.0	0.2	21.2
5	Highvale	Sb B	13.5	10.7	39.6	60.4	75.0	4.4	1.1	0.2	19.3
6	Forestburg	Sb C	9.6	17.5	43.3	56.7	73.4	5.0	1.5	0.9	19.2
7	Vesta Mine	Sb C	5.0	12.2	41.9	58.1	74.4	5.0	1.5	0.6	18.5
8	Heatburg–Fording	Sb B	6.2	17.4	37.9	62.1	74.5	4.3	1.0	0.4	19.8
9	Sheerness	Sb C	13.0	10.9	43.2	56.8	73.7	4.6	1.7	0.8	18.2
10	Atlas Mine–East Coulee	Sb B	18.8	8.5	42.1	56.9	76.3	5.1	1.7	0.7	16.2

Table 5. Petrographic analyses of Alberta Plains coals

Sample no.	Location	ASTM rank	Volume percent maceral composition (mineral matter free)					Vitrinite Reflectance (random) %
			Vitrinite (huminite)	Liptinite	Semifusinite	Fusinite	Other Inertinites	
1	Smoky Tower	Sb A	70.0	6.4	8.6	4.3	10.7	0.51
2	Egg Lake	Sb C	92.4	4.2	0.8	0.4	2.2	0.33
3	Starkey	Sb C	91.5	5.8	1.5	0.6	0.6	0.33
4	Wabamun	Sb B	65.0	6.0	15.0	3.0	11.0	0.36
5	Highvale	Sb B	74.6	2.1	22.2	1.1	nil	0.48
6	Forestburg	Sb C	92.5	3.8	0.6	1.9	1.2	0.39
7	Vesta	Sb C	73.3	0.3	8.9	3.3	14.1	0.43
8	Heatburg	Sb B	74.8	1.0	9.6	2.1	12.5	0.44
9	Sheerness	Sb C	92.7	5.3	1.2	0.4	nil	0.42
10	East Coulee	Sb B	91.9	4.9	0.6	1.4	1.2	0.46

Table 6. Chemical composition of lignites

Sample no.	Location	Proximate analysis (%) Moisture	Ash (dry)	Volatile matter (d.a.f.)	Fixed Carbon (d.a.f.)	Ultimate analysis (%) (dry & ash free) Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen
A	Klimax, Sask.	8.8	17.0	43.2	56.8	73.3	4.5	1.2	1.2	19.8
B	Bienfait, Sask.	6.3	7.1	45.2	54.8	73.2	4.6	1.2	1.1	19.9
C	North Dakota	29.1	16.4	50.1	49.7	70.8	4.6	1.2	2.8	20.6
D	Texas	5.2	11.9	51.3	48.7	72.7	5.2	1.5	1.3	19.3

Table 7. Petrographic analysis of lignites

Sample no.	Location	Volume percent maceral composition (mineral matter free) Vitrinite (huminite)	Liptinite	Semifusinite	Fusinite	Other Inertinites	Vitrinite Reflectance (Random) %
A	Klimax, Sask.	83.1	0.4	3.9	1.8	10.8	0.36
B	Bienfait, Sask.	69.0	13.0	9.0	2.0	7.0	0.36
C	North Dakota	60.3	5.3	22.1	12.3	nil	0.24
D	Texas	60.9	7.6	10.9	10.5	10.1	0.33

Table 8.
Comparison of petrographic analyses of a subbituminous coal* (Smoky Tower— Sb A) conducted by five operators

Maceral	Volume percent maceral composition #1	#2	#3	#4	#5	Average
Vitrinite (huminite)	64	68	64	64	64	65
Liptinite	6	5	5	4	8	6
Semifusinite	9	7	8	10	8	8
Fusinite	5	4	3	2	5	4
Other Inertinites	11	13	7	8	10	10
Mineral matter	5	3	13	10	5	7

* Same polished coal pellet analyzed on the same day using same microscope

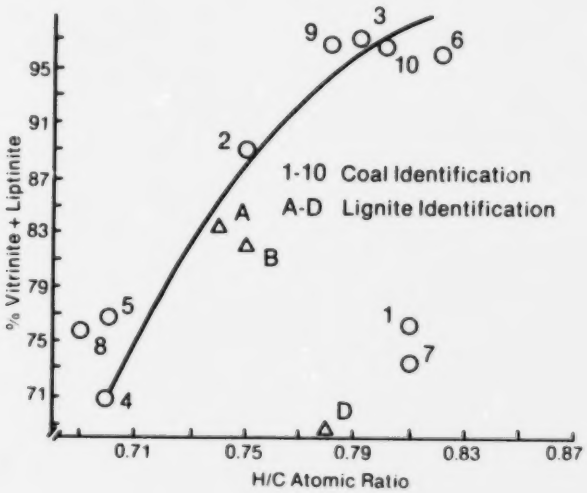


Figure 5. Relationship between vitrinite-liptinite macerals and H/C atomic ratio

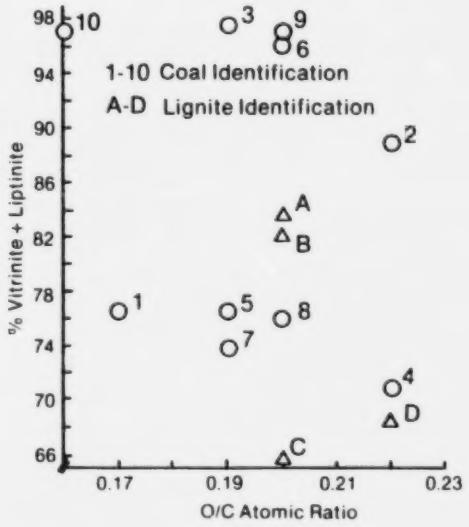


Figure 6. Relationship between vitrinite-liptinite macerals and O/C atomic ratio

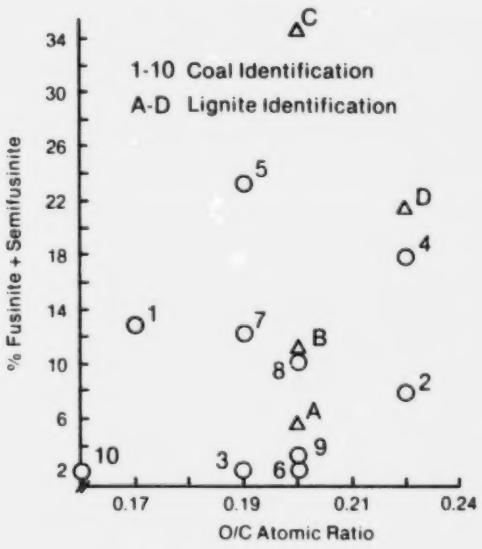


Figure 8. Relationship between fusinite-semifusinite macerals and O/C atomic ratio

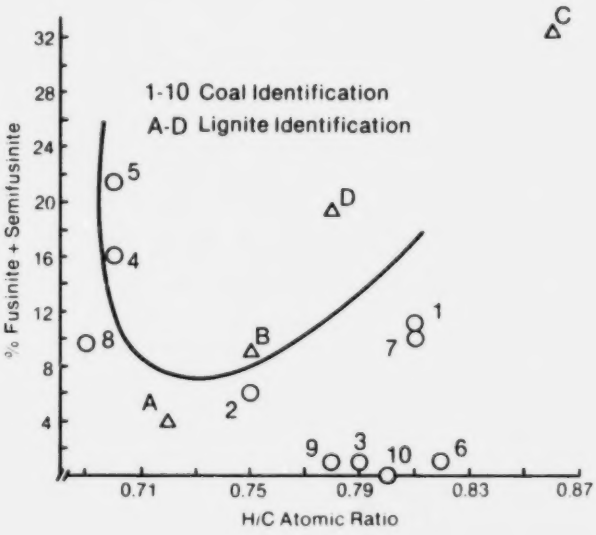


Figure 7. Relationship betwen fusinite-semifusinite macerals and H/C atomic ratio

Reflectance of the vitrinite (huminite) component

Reflectance of vitrinite increases progressively with increasing rank (van Krevelen, 1962) and the measurement of reflectance of the coal maceral vitrinite has become a routine procedure in characterization of coal rank. Reflectance data of vitrinite (huminite) in subbituminous coals and lignites are given in table 9. The reflectance reported is the average of 100 random measurements using a polarized light source. The data match ASTM rank classification, based on reflectance of vitrinite (Stach *et al.*, 1982) and can clearly differentiate subbituminous A coal from subbituminous B or subbituminous C coals. Among the lignites, the degree of coalification based on reflectance of huminite (vitrinite) follows the order:

Saskatchewan lignites→Texas lignite→North Dakota lignite

Table 9. Random oil reflectance of subbituminous coals and lignites

Subbituminous Coals		Lignites	
Sample Identification	Random reflectance (%) of Vitrinite (polarized)	Sample Identification	Random reflectance (%) of Vitrinite (polarized)
Smoky Tower	0.51 ± 0.05	Klimax	0.36 ± 0.03
Egg Lake	0.33 ± 0.04	Bienfait	0.36 ± 0.02
Starkey	0.33 ± 0.03	North Dakota	0.24 ± 0.04
Wabamun	0.38 ± 0.02	Texas	0.33 ± 0.01
Highvale	0.48 ± 0.04		
Forestburg	0.39 ± 0.04		
Vesta	0.43 ± 0.04		
Heatburg	0.44 ± 0.04		
Sheerness	0.42 ± 0.03		
East Coulee	0.46 ± 0.03		

Reflectance of vitrinite contained in a sample is not constant, but shows a scatter of ~0.05 percent reflectance about the mean value, following more or less a Gaussian curve. The reflectance distribution for three subbituminous coals (Smoky Tower Sb A; Highvale Sb B; Vesta Sb C) and two lignites (North Dakota and Texas) are shown in the form of ½-V type histograms in figures 9 through 13, and the reflectance percent distribution curves are shown in figures 14 through 18. Each ½-V type represents a reflectance range of 0.05 percent; for example, all readings in an analysis between 0.40 and 0.44 percent represent ½-V type class, designed by the mid-point expression 4.25 and are assigned to V-stage 4. Reporting results in this manner is useful to demonstrate the contribution of coals of different ranks in a blend (International Committee for Coal Petrography, 1971).

To compare frequency distributions of reflectance of vitrinite components of subbituminous A, subbituminous B and subbituminous C coals and two U.S. lignites more rigidly, ½-V type distribution curves and percent distribution curves were superimposed on each other for coals in figures 19 and 20 and for lignites in figures 21 and 22.

Reflectance versus chemical composition

Present standard systems for classification of coals by rank are intended mainly as guides for utilization behavior, and there is a need for more widespread acceptance and usage of an objective rank criterion based more firmly on physical/chemical characteristics of a coal. McCartney and Teichmuller (1972) have investigated the utility of reflectance of the coal maceral vitrinite as a criterion of degree of coalification, and have reported excellent correlations between reflectance of the vitrinite and volatile matter of carbon content for high rank bituminous coals. The data from these investigations of subbituminous coals and lignites show a good relation of reflectance of vitrinite to carbon content (figure 23) but do not establish any significant correlation between the reflectance and volatile matter. McCartney and Teichmuller (1972) also acknowledge this fact. van Krevelen (1962), Davis, (1965) and McCartney and Teichmuller (1972) reported a linear correlation between reflectance and carbon content for low rank coals (70 to 80 percent dry-ash-free carbon). For quick comparison, their data are included in figure 23.

Figure 9. Reflectance histogram of Smoky Tower coal

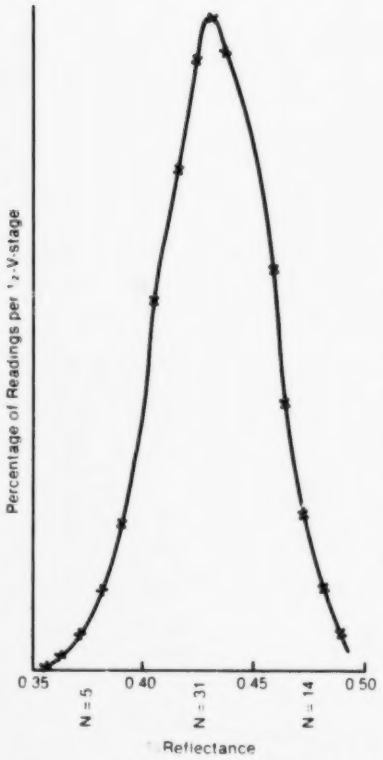


Figure 10. Reflectance histogram of Highvale coal

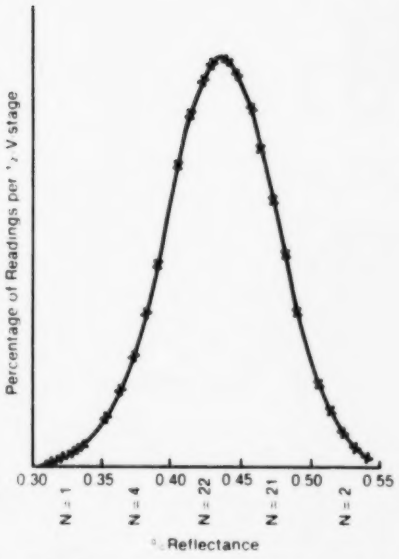


Figure 11. Reflectance histogram of Vesta mine coal

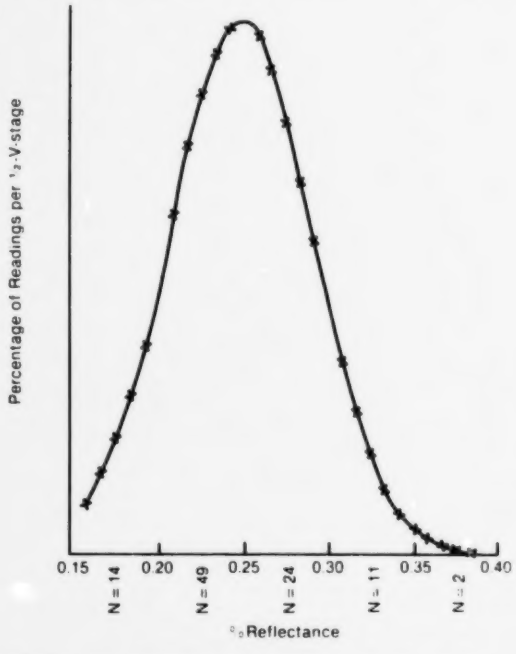


Figure 12. Reflectance histogram of North Dakota lignite

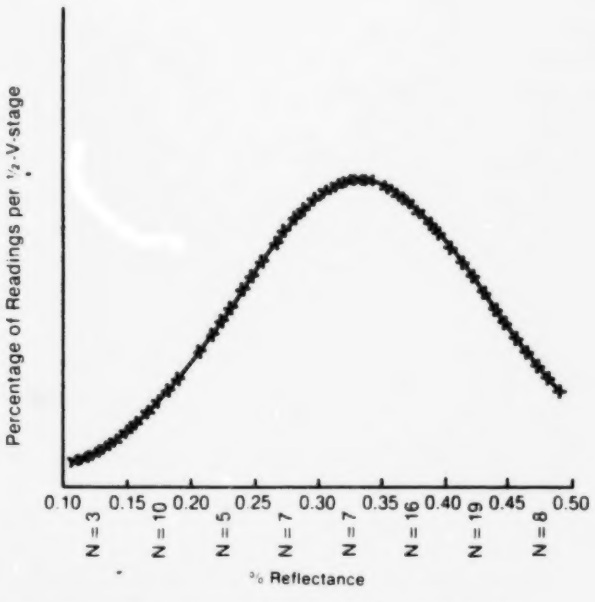


Figure 13. Reflectance histogram of Texas lignite

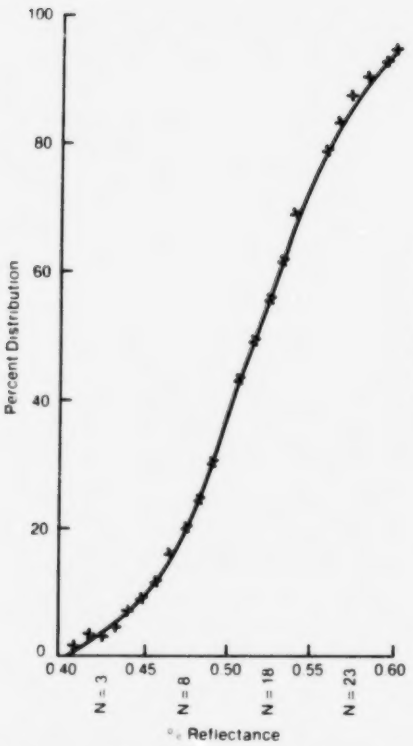


Figure 14. Reflectance percent distribution curve of Smoky Tower coal

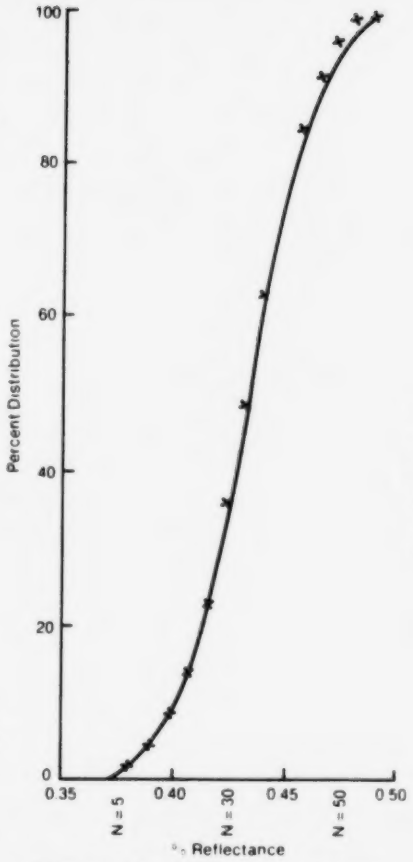


Figure 15. Reflectance percent distribution curve of Highvale coal

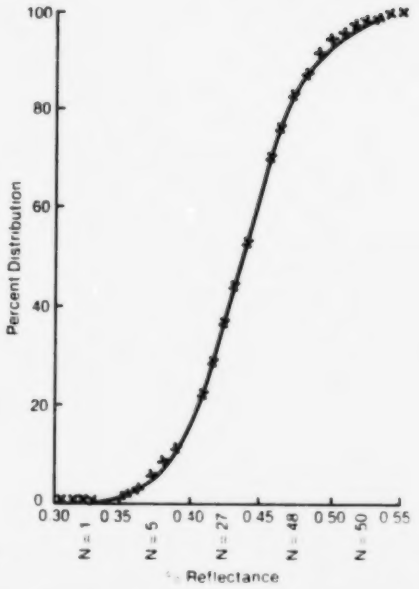


Figure 16. Reflectance percent distribution curve of Vesta mine coal

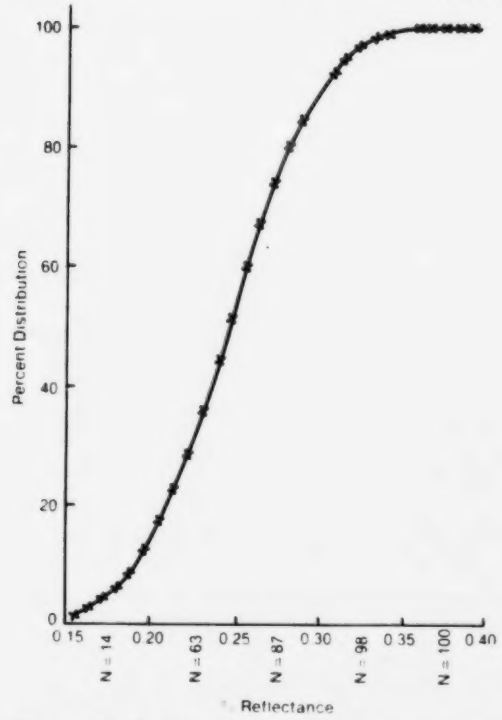


Figure 17. Reflectance percent distribution curve of North Dakota lignite

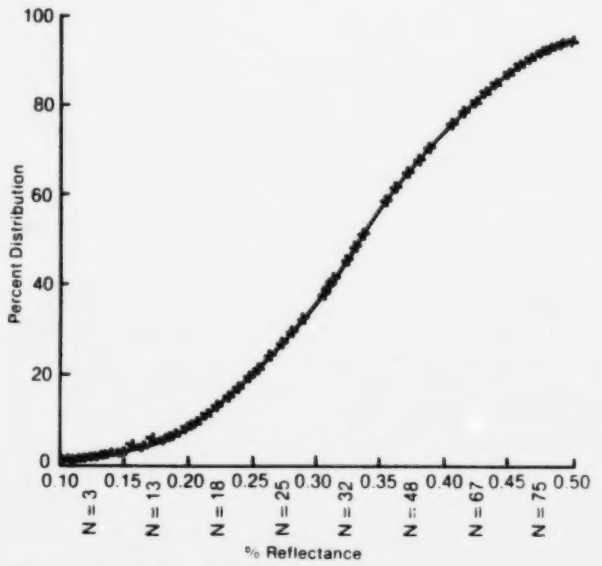


Figure 18. Reflectance percent distribution curve of Texas lignite

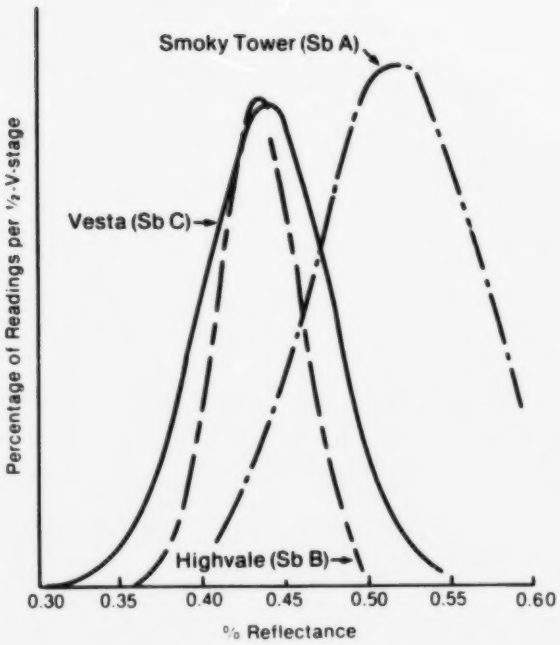


Figure 19. Reflectance frequency distribution diagram for subbituminous coals

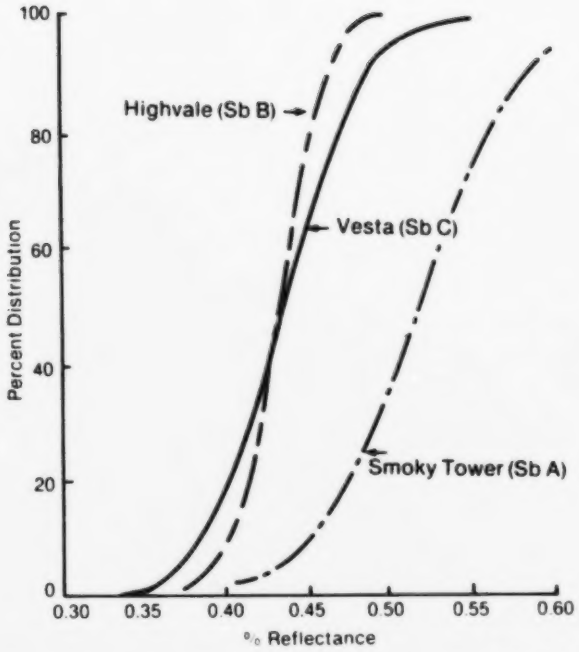


Figure 20. Reflectance percent distribution curve for subbituminous coals

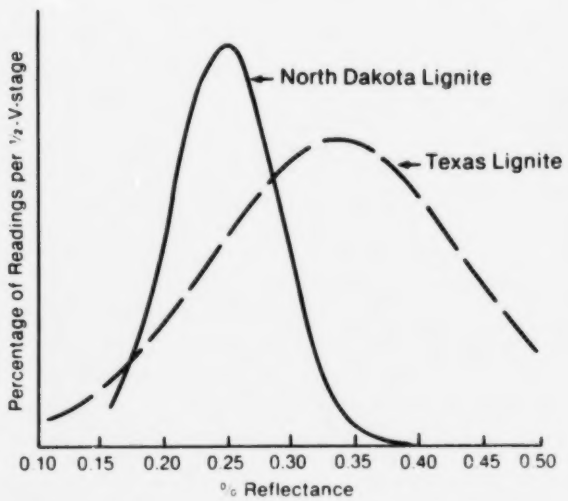


Figure 21. Reflectance frequency distribution diagram for U.S. lignites

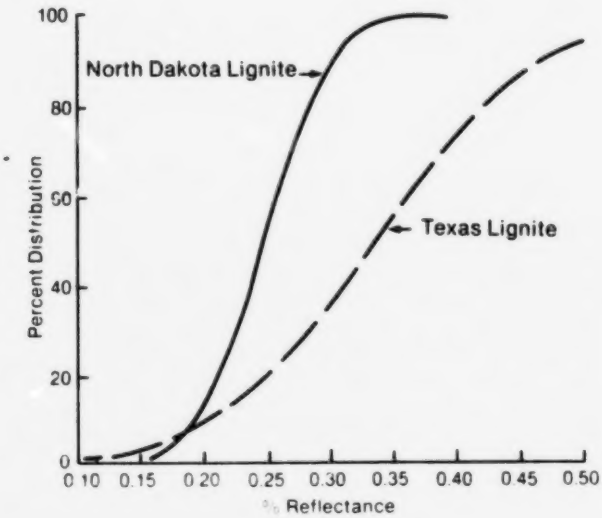


Figure 22. Reflectance percent distribution curve for U.S. lignites

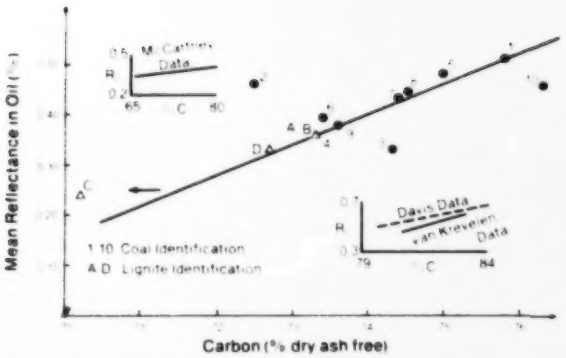


Figure 23. Relationship between mean reflectance of vitrinite and carbon content

Photomicrographs

It is a normal practice with coal petrologists to communicate through photomicrographs. The photomicrographs provide some information on the cellular characteristics and granularity of different macerals in coals. Reflectance measurement, however, is the only practical method to determine their rank or degree of coalification.

Plates 1 through 5 show typical photomicrographs for three Alberta subbituminous coals and two U.S. lignites, which indicate the manner in which different coal macerals and mineral matter are distributed. It becomes evident from the photomicrographs that Alberta subbituminous coals nave a low concentration of the macerals of the liptinic group. They are almost free from pyrite and the mineral matter is distributed irregularly.

Comparing the photomicrographs of two U.S. lignites indicates that:

a. Texas lignite is richer in liptinite but poorer in semifusinite than North Dakota lignite, and
b. the Texas lignite contains some inertinite (inertodetrinite?) other than fusinite.

Petrographic composition of density fractions

As coal science progresses, the need for pure coal macerals becomes increasingly obvious. The float-and-sink separation technique, commonly applied for separation of coal macerals, simply provides density fractions enriched in specif'c maceral groups. From the petrographic composition of the density fractions, advantage can be taken to evaluate the contribution of different macerals for coal in the production of liquid hydrocarbons.

The float-and-sink separation technique, using mixtures of halogenated and nonhalogenated hydrocarbons (cyclohexane, tetrachlorcethylene, carbon tetrachloride and bromoform) of different densities (in the range 1.35 to 1.60 gcm $^{-3}$), was employed to obtain different density fractions of subbituminous coals and lignites.

Approximately 100 g of a coal or lignite (oven-dried; minus 40 mesh) were immersed in 1 L of organic solvent with a density 1.35 gcm^{-3} at room temperature. The mixture was stirred for a few hours, using a magnetic stirrer and then allowed to stay stationary overnight. The float and sink portions were removed separately and dried thoroughly in an oven (1-2 mm Hg) at 40 to 45°C for 24 hours. The sink material was reimmersed in a fresh solution of a higher density and the procedure was repeated. A schematic procedure for obtaining different density fractions of lignite is given in figure 24.

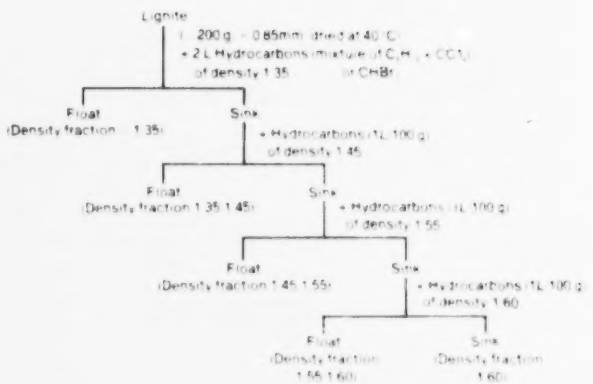


Figure 24. Schematic procedure for separation of density fraction of lignite

The chemical analyses of density fractions of two U.S. lignites and two subbituminous coals (Highvale Sb B and Vesta Sb C) are given in tables 10 and 11. As expected, the ash content increases progressively with an increase in fraction densities and various fractions differ considerably in their chemical compositions.

Petrographic composition and average random reflectance for the density fractions of two U.S. lignites (North Dakota and Texas) and two subbituminous coals (Highvale Sb B and Vesta Sb C) are given in tables 12 through 15. It is apparent from the petrographic analyses that:

a. For density fractions < 1.35 and 1.35-1.45, the combined vitrinite (huminite) and liptinite contents are higher than the respective values for higher density fractions. These data agree with chemical analyses (tables 10 and 11), since liptinite is normally distinguished by a relatively high hydrogen content. Careful examination of the data (tables 10 to 15) supports the accepted view that the vitrinite (huminite) contains relatively more oxygen, the liptinite more hydrogen and inertinite (fusinite and other inerts) more carbon.
b. There is no significant difference in the reflectance of different density fractions for either of the subbituminous coals or lignites studied. This seems to result from the fact that the mean reflectance of a vitrinite (huminite) in a given coal is largely uninfluenced by variation in petrographic composition.

The photomicrographs (plates 1 and 2) give a general idea of how different macerals are distributed in different density fractions of lignites.

Table 10. Chemical analysis of density fractions of U.S. lignites

	North Dakota lignite						Texas lignite					
	Dry Basis						Dry Basis					
Fraction density (g cm ^{3})	Wt. % of density fraction	% Ash	% C	% H	% N	% (O + S + Halogens)	Wt. % of density fraction	% Ash	% C	% H	% N	% (O + S + Halogens)
Composite	-	17.1	58.7	3.8	1.0	19.4	-	12.8	63.4	4.5	1.3	18.0
< 1.35	0.6	4.7	69.4	5.0	0.8	20.1	15.2	4.7	69.8	5.1	0.8	19.6
1.35-1.45	40.3	8.3	65.0	4.6	1.0	21.1	73.4	6.9	63.2	4.6	1.2	24.1
1.45-1.55	33.7	11.2	61.8	3.9	1.1	22.0	5.7	9.5	59.9	4.2	1.2	25.2
1.55-1.60	7.8	13.8	60.7	3.9	1.1	20.5	0.7	15.0	56.3	4.1	1.3	23.3
> 1.60	17.6	28.9	47.5	2.9	0.9	19.8	5.0	29.5	47.4	3.6	1.0	18.5

Table 11. Chemical analysis of density fractions of subbituminous coals

	Highvale coal (subbituminous B)						Vesta coal (subbituminous C)					
	Dry Basis						Dry Basis					
Fraction density (g cm ^{3})	Wt. % of density fraction	% Ash	% C	% H	% N	% (O + S + Halogens)	Wt. % of density fraction	% Ash	% C	% H	% N	% (O + S + Halogens)
Composite	-	10.7	67.0	3.9	1.0	17.4	-	12.2	65.3	4.4	1.3	16.8
< 1.35	13.8	4.0	71.7	4.4	0.8	19.1	20.6	5.3	69.6	4.3	1.6	19.2
1.35-1.45	39.9	5.3	70.8	4.2	0.9	18.8	20.7	6.3	69.2	4.3	1.6	18.6
1.45-1.55	25.8	8.5	68.8	3.7	1.0	18.0	43.7	10.3	65.9	4.2	1.6	18.0
1.55-1.60	7.8	11.7	66.4	3.4	0.9	17.6	4.0	17.5	60.4	3.9	1.7	16.5
> 1.60	13.3	42.9	39.1	2.5	0.7	14.8	5.0	52.5	33.9	2.5	0.9	10.2

Table 12. Maceral analysis of density fractions of North Dakota lignite

Fraction density (g cm ^{3})	Volume % Maceral Composition						Average random reflectance of huminite
	Huminite	Liptinite	Semifusinite	Fusinite	Other inerts	Mineral matter	
Composite	52.9	4.6	19.4	10.8	nil	12.3	0.24
< 1.35	69.7	4.3	13.7	2.3	nil	10.0	0.21
1.35-1.45	87.7	6.3	4.0	nil	nil	2.0	0.26
1.45-1.55	64.0	3.0	24.7	4.0	nil	4.3	0.26
1.55-1.60	53.0	2.6	28.5	5.3	nil	10.6	0.28
> 1.60	15.7	2.0	28.0	23.7	nil	30.6	0.28
	Volume % Maceral Composition (mmf)						
Composite	60.3	5.3	22.1	12.3	nil		
< 1.35	77.4	4.8	15.2	2.6	nil		
1.35-1.45	89.5	6.4	4.1	nil	nil		
1.45-1.55	66.9	3.1	25.8	4.2	nil		
1.55-1.60	59.3	2.9	31.9	5.9	nil		
> 1.60	22.6	2.9	40.3	34.2	nil		

Table 13. Maceral analysis of density fractions of Texas lignite

Fraction density (g cm ^{3})	Volume % Maceral Composition						Average random reflectance of huminite
	Huminite	Liptinite	Semifusinite	Fusinite	Other inerts	Mineral matter	
Composite	56.0	7.0	10.0	9.7	9.3	8.0	.34
< 1.35	65.7	8.3	4.0	6.0	7.7	8.3	.36
1.35-1.45	70.7	8.3	2.0	3.0	12.7	4.7	.31
1.45-1.55	45.7	2.7	3.0	0.7	3.7	42.0	.30
1.55-1.60	34.0	3.7	11.3	12.3	9.7	29.0	.29
> 1.60	26.3	3.3	10.3	9.7	6.0	44.4	.31
	Volume % Maceral Composition (mmf)						
Composite	60.9	7.6	10.9	10.5	10.1		
< 1.35	71.6	9.1	4.4	6.5	8.4		
1.35-1.45	79.7	9.4	2.3	3.3	5.3		
1.45-1.55	82.0	4.7	5.4	1.2	6.7		
1.55-1.60	47.9	5.2	15.9	17.3	13.7		
> 1.60	47.3	5.9	18.5	17.5	10.8		

Table 14. Maceral analysis of density fractions of Highvale coal (subbituminous B)

Fraction density (g cm ^{3})	Volume % Maceral Composition						Average random reflectance of huminite
	Huminite	Liptinite	Semifusinite	Fusinite	Other inerts	Mineral matter	
Composite	65.0	1.2	19.6	2.0	1.2	11.0	.48
< 1.35	93.6	0.7	2.7	nil	0.7	2.3	.46
1.35-1.45	72.0	6.2	6.8	2.2	9.0	3.8	.43
1.45-1.55	47.5	3.5	35.5	3.1	4.5	5.9	.43
1.55-1.60	42.7	1.7	23.8	16.3	4.5	11.0	.43
> 1.60	22.4	0.5	16.5	9.8	5.1	45.7	.44
	Volume % Maceral Composition (mmf)						
Composite	73.0	1.3	22.1	2.3	1.3		
< 1.35	95.9	0.7	2.8	nil	0.6		
1.35-1.45	74.9	6.3	7.1	2.3	9.4		
1.45-1.55	50.5	3.7	37.6	3.4	4.8		
1.55-1.60	48.0	1.9	26.7	18.3	5.1		
> 1.60	41.3	0.9	30.4	18.0	9.4		

Table 15. Maceral analysis of density fractions of Vesta mine coal (subbituminous C)

Fraction density (g cm ^{3})	Volume % Maceral Composition						Average random reflectance of huminite
	Huminite	Liptinite	Semifusinite	Fusinite	Other inerts	Mineral matter	
Composite	74.2	2.6	5.2	2.3	10.5	5.2	.41
< 1.35	87.9	5.2	2.7	1.0	1.4	1.8	.43
1.35-1.45	88.3	2.7	2.0	1.0	3.0	3.0	.46
1.45-1.55	77.5	1.7	6.4	2.7	5.2	6.5	.44
1.55-1.60	67.0	1.3	5.3	4.9	9.5	12.0	.47
> 1.60	38.4	0.8	2.7	3.9	7.2	47.0	.45
	Volume % Maceral Composition (mmf)						
Composite	78.2	2.7	5.5	2.5	11.1		
< 1.35	89.5	5.3	2.8	1.0	1.4		
1.35-1.45	91.0	2.8	2.1	1.0	3.1		
1.45-1.55	82.8	1.9	6.8	2.9	5.6		
1.55-1.60	76.2	1.5	6.0	5.6	10.7		
> 1.60	72.5	1.5	5.0	7.3	13.7		

Liquefaction behavior and maceral composition

An aim of the research work of coal petrologists in the field of coal liquefaction is to predict the yield of liquid and/or gaseous products starting from a given rank of the coal and a given maceral composition. Several correlations have been predicted with limited data (Yarzab *et al.*, 1980) but none have provided a satisfactory answer.

Alberta Research Council's coal liquefaction data, with limited number of subbituminous coal samples, do not permit development of mathematical correlations between conversion yields and coal characteristics. Nevertheless, regression equations developed by Yarzab *et al*, (1980) were tested on short-residence-time liquefaction data of three subbituminous coals (Parkash *et al.*, 1982). Only one of these equations appeared to work on these data (table 16):

$$\% \text{ Conversion} = 1.61\, S_t + 0.20\, TRM + 1.79\, VM - 33.7$$

where S_t = wt % (dry) total sulfur
TRM = vol % (mmf) content of relative macerals (vitrinite and liptinite)
VM = wt % (mmf) volatile matter

Table 16. Liquefaction behavior dependence on coal characteristics

	% Total Conversion (d.a.f. coal)	
Coal	Experimental	Calculated from Regressions Eq. Conv. = $1.61S_t$ + 0.20TRM + 1.79VM -33.7
Smoky Tower	57	60
Highvale	53	53
Vesta	66	57

Liquefaction data for density fractions of U.S. lignites (North Dakota and Texas) in relation to petrographic composition have indicated (Parkash *et al.*, 1981) that, in both lignites, the total conversion decreases systematically with increase in density fractions (figures 25 and 26). This is likely due to the decrease in the combined huminite (vitrinite) and liptinite contents—termed reactives—in the high density fractions. On plotting total conversion of parent lignites and their density fraction against their huminite contents, some rectilinear correlation becomes apparent (figure 27). For North Dakota lignite, this correlation is far more linear than for Texas lignite. Since for the density fractions separated from North Dakota lignite the ratio of huminite to remaining macerals varies considerably from fraction to fraction, one can draw a tentative hypothesis that the contribution of the remaining macerals (semifusinite, fusinite, other inertinites and liptinite) to the solubility yield, for the North Dakota lignite, is minimal.

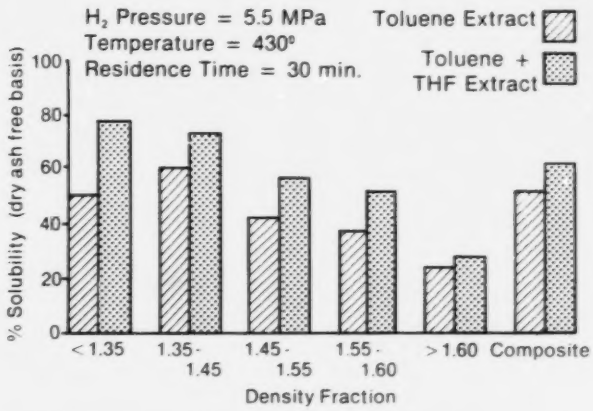


Figure 25. Solubilities of North Dakota lignite

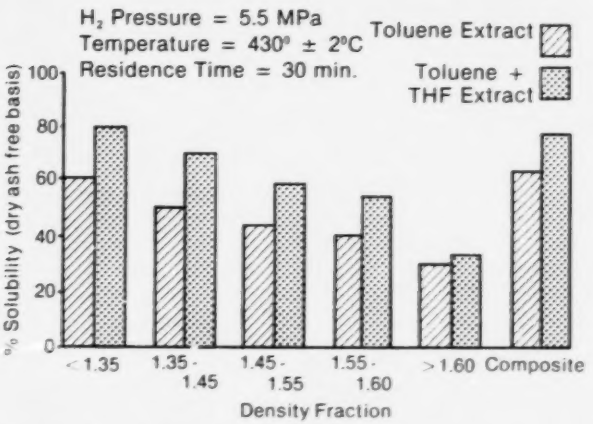


Figure 26. Solubilities of Texas lignite

In North Dakota lignite the macerals other than huminite essentially do not contribute to or modify the liquefaction process. The solubility on liquefaction of the composite sample (62.1 percent) is very close to the cumulative solubility (59.2 percent) calculated from the solubility percentages determined for the individual density fractions. The same procedure applied for Texas lignite showed, as expected, a much larger spread, 78.1 percent and 70.2 percent respectively.

It is premature to advocate a conversion factor for any maceral group to predict the liquefaction behavior of a coal. Shibaoka *et al.* have fairly intensively studied the role of different macerals from high-volatile bituminous coals during hydrogenation. According to their findings (Shibaoka, 1981; Shibaoka *et al.*, 1981), two different materials can form from vitrinite macerals—lamellar or fibrous solid material, and plastic material vitroplast. The first type is derived from ungelled cellular material, and the second from humic gel and highly gelled material.

A schematic of the behavior of huminite and vitrinite macerals and submacerals, when hydrogenated with tetralin at lower temperatures (300 to 380°C), is given in figure 28.

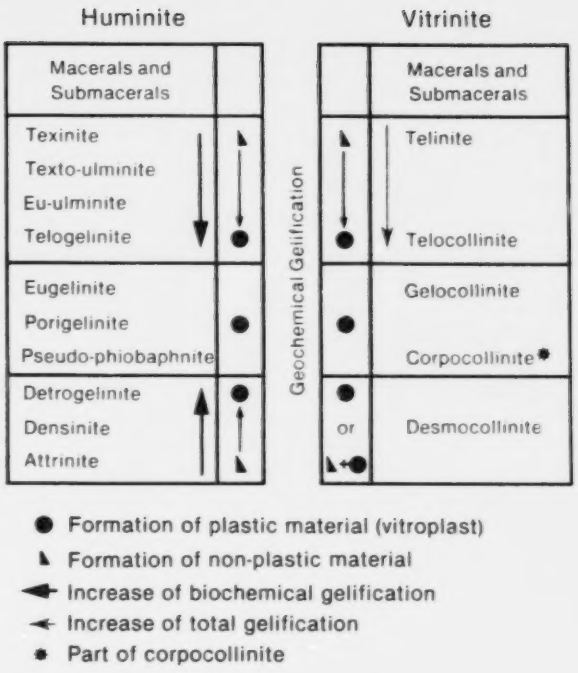


Figure 28. Behavior of huminite and vitrinite macerals and submacerals upon hydrogenation

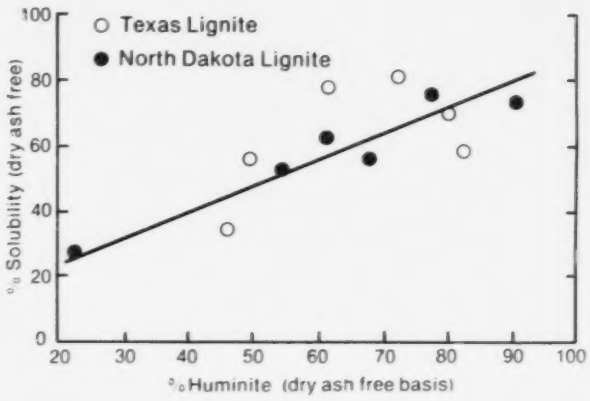


Figure 27. Relationship between huminite contents and lignite solubility

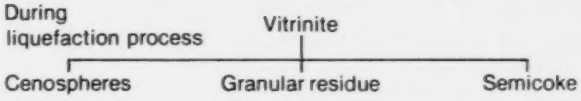
Petrographic analysis of liquefied coal residues

Residue is the tetrahydrofuran (THF) insoluble product obtained from liquefied coal. Petrographic analysis of residues produced during hydrogenation is a subject of very recent investigations. Optical examination of residues is a very practical approach to selecting optimum process conditions for coal liquefaction.

Petrographic analysis of residues obtained from the three coals in I.G. Farben-type process and CO-steam type process are given in table 17. Plates 3 through 5 show photomicrographs of the coals and their residues.

General conclusions are drawn from optical examination of liquefied coal.

1. Several basic materials originating from the organic maceral constituent of feed coal have been identified.
2. Vitrinite has changed to:
   a. plastic solid cenospheres (high gray spheres), which are believed to result from gas production by thermal cracking;
   b. granular residue, which is unreacted and finely dispersed vitrinite that formed after the dissolution of vitrinite and liptinite;
   c. semicoke, which originated from liquefied vitrinite under severe reaction conditions.



3. Semifusinite has solubilized partially and the unreacted portion appears in the residue. It has undergone mechanical grinding and, as a result, a distinction between semifusinite and fusinite cannot be obtained.

Table 17. Petrographic analysis of liquefied coal residues

	I.G. Farben Process (P = 6.9 MPa; T = 450°C; t = 10 min)			CO-Steam Process (P = 5.5 MPa; T = 400°C; t = 10 min)		
	Percent Volume (mineral-matter-free) in residues from:			Percent Volume (mineral-matter-free) in residues from:		
Component	Smoky Tower Coal	Highvale Coal	Vesta Coal	Smoky Tower Coal	Highvale Coal	Vesta Coal
Vitrinite	13.4	12.1	9.6	10.4	21.8	8.9
Semifusinite	1.5	5.5	1.9	2.1	1.1	0.2
Fusinite	0.2	0.6	nil	1.1	0.6	nil
Granular Residue*	72.8	71.6	75.8	80.5	67.9	83.2
Semicoke	3.1	6.0	7.8	4.0	4.1	1.0
Bright Fragments	8.8	4.2	4.9	1.9	4.5	6.7
Cenospheres	0.2	nil	nil	nil	nil	nil
Average Random Reflectance of Vitrinite	1.39	1.56	1.17	1.14	1.02	1.07

*Particles less than 10 mm not identifiable as individual components; includes both maceral and mineral-derived materials.

4. Fusinite appears to have merged with granular residue after simple mechanical fining and remains undistinguished.
5. Unreacted or even partially reacted liptinite is not detected.
6. Mineral matter has dispersed in small fines.
7. Fusinite, dispersed clays, quartz, pyrrhotite and carbonate stay in the residual components and are not reactive constituents.
8. I.G. Farben-type process residue contains more semicoke compared to CO-steam type process residue.
9. Unreacted vitrinite is dispersed uniformly in the I.G. Farben process residue, while CO-steam residue contains clusters of unreacted vitrinite.
10. Quantitive microscopic analysis of residue components gives the clue to evaluate the partial reactivity of maceral consitituents such as semifusinite, and to make a subjective comparison of the effectiveness of the different run conditions in the liquefaction processes.

Strehlow *et al.* (1978) suggest that submicron-sized mineral components within vitrinite may influence the conversion of coal into liquids. Their data show that the surface area of the submicron-sized mineral matter is significantly large and can be expected to induce a variety of catalytic reactions in coal liquefaction.

Petrological methods are advancing in characterizing the behavior of coal during hydrogenation, but it would be premature to evaluate with confidence the liquefaction behavior of a particular coal from the comparative study of maceral composition with those of the hydrogenation residues. Much more research work is needed before a complete understanding is possible of all transitional stages during hydrogenation of different kinds of coal and the interaction between coal and various catalysts.

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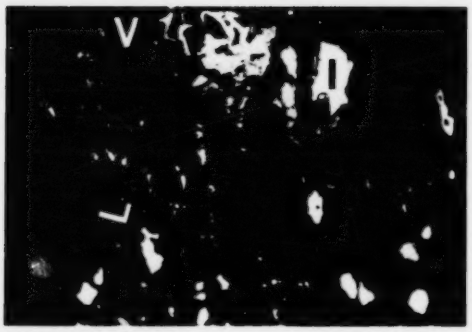
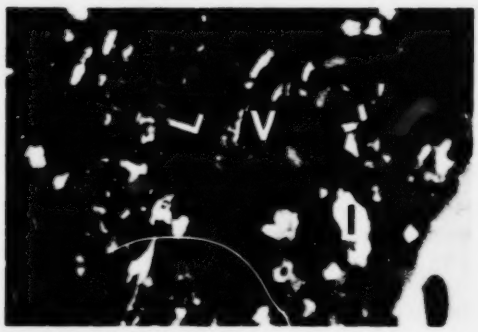
Plate 1

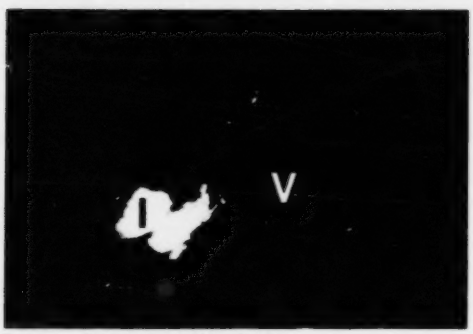
Photomicrographs of North Dakota lignite and its density fractions x 320



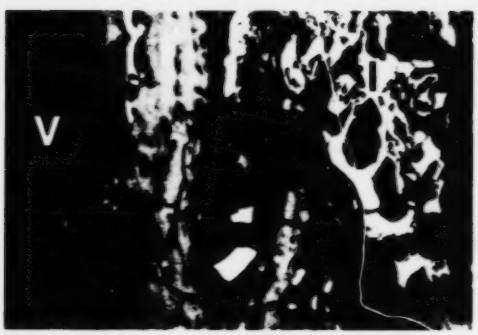
Composite lignite



Density fraction < 1.35



Density fraction 1.35 - 1.45



Density fraction 1.45 - 1.55

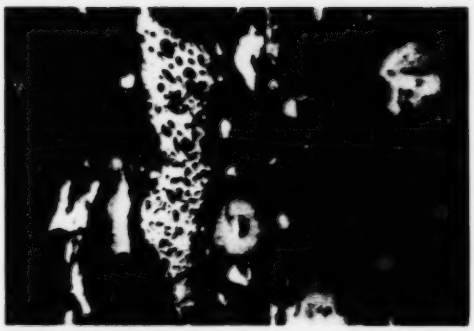
Density fraction 1.55 - 1.60

Density fraction > 1.60

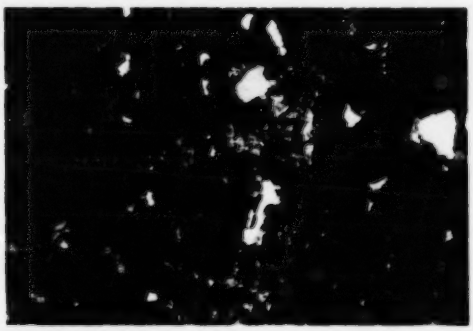
V = Vitrinite; L = Liptinite; I = Inertinite

Plate 2

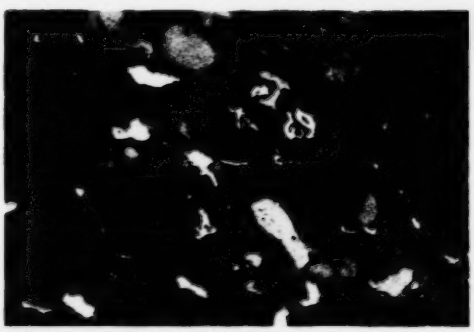
Photomicrographs of Texas lignite and
its density fractions x 320



Composite lignite

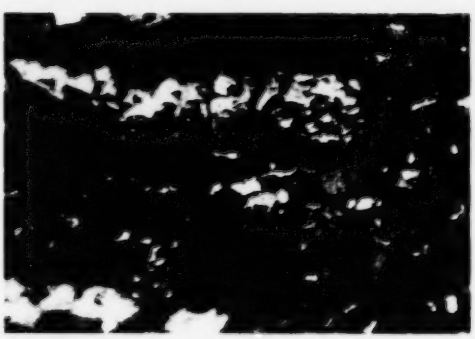


Density fraction < 1.35

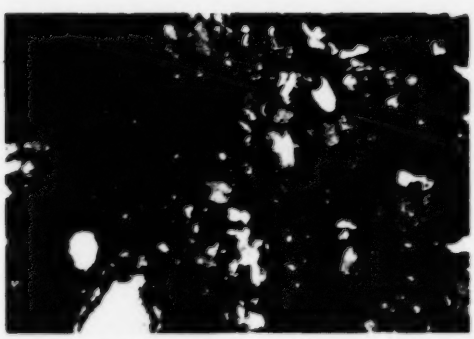


Density fraction 1.35 - 1.45

Density fraction 1.45 - 1.55



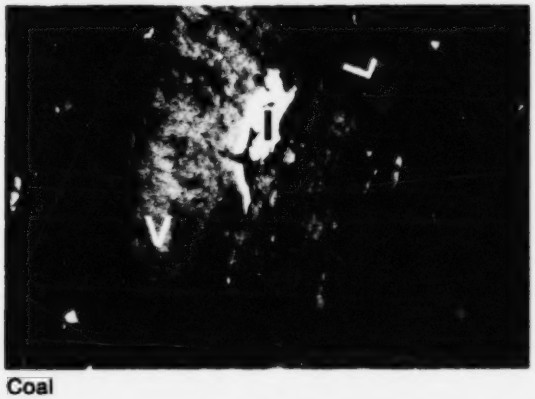
Density fraction 1.55 - 1.60



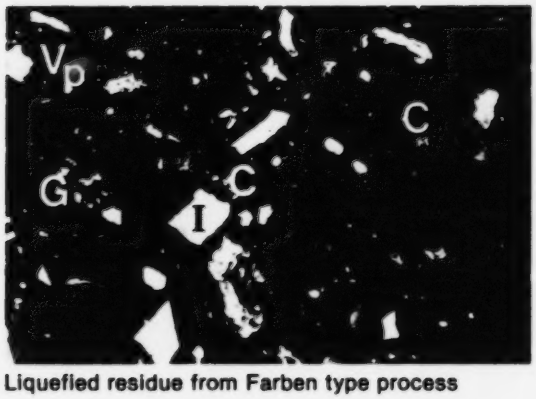
Density fraction > 1.60

Plate 3

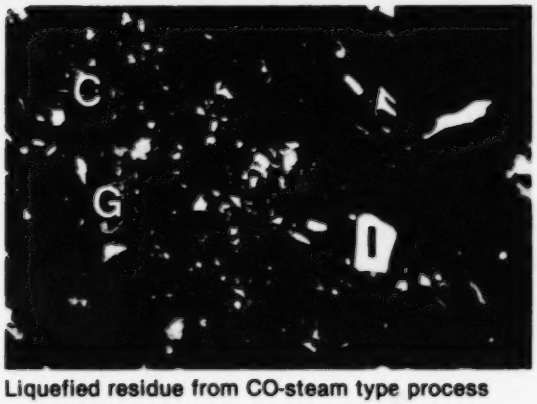
Photomicrographs of Smoky Tower (subbituminous A) coal and liquefied residues x 480



Coal



Liquefied residue from Farben type process

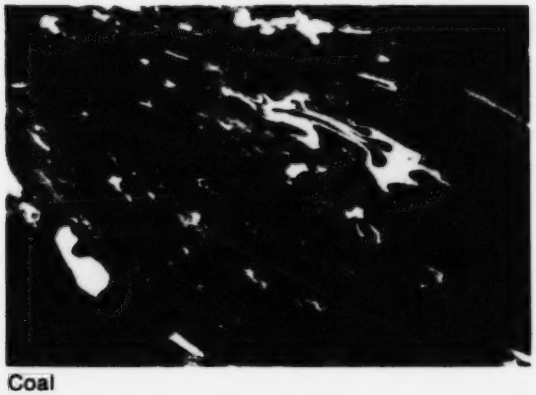


Liquefied residue from CO-steam type process

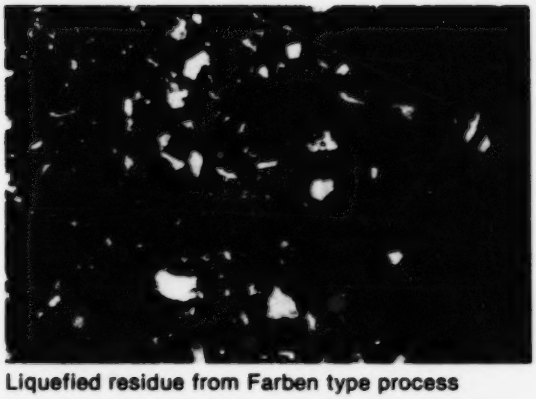
V = Vitrinite; Vp = Vitroplast; I = Inertinite; C = Coke/semicoke; G = Granular residue

Plate 4

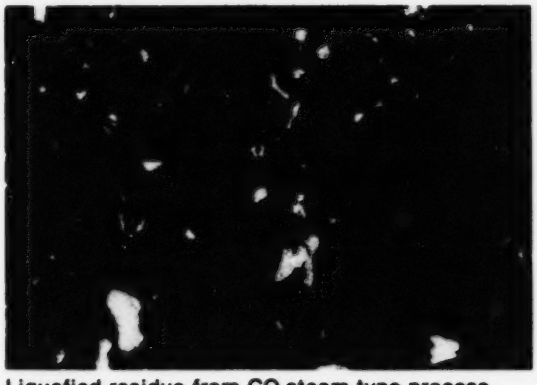
Photomicrographs of Highvale (subbituminous B) coal and liquefied residues x 480



Coal



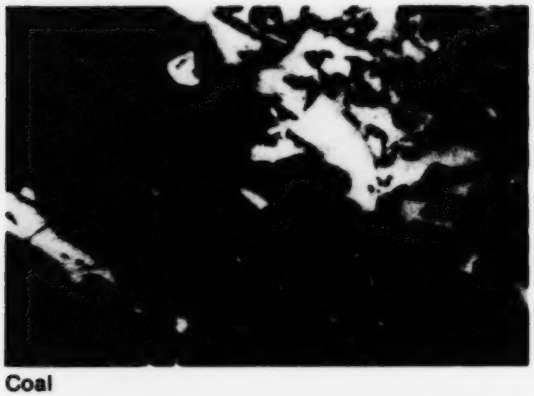
Liquefied residue from Farben type process



Liquefied residue from CO-steam type process

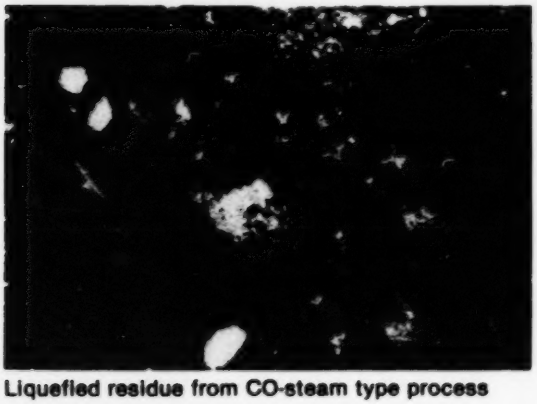
Plate 5

Photomicrographs of Vesta (subbituminous C) coal and liquefied residues x 480



Coal

Liquefied residue from Farben type process



Liquefied residue from CO-steam type process